BULK SYNTHESIS OF GRAPHENE NANOSHEETS

A THESIS SUBMITTED IN THE PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF BACHELOR OF TECHNOLOGY

By

Sohan Choudhuri

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Under the Guidance of

Dr. Shantanu Behera

NATIONAL INSTITUTE OF TECHNOLOGY ROURKELA

CERTIFICATE

This is to certify that the thesis entitled, **"Bulk Synthesis of Graphene Nanosheets"** is the bonafide work of Mr. **Sohan Choudhuri (Roll no. 108CR053)** in partial fulfilments for the requirements for the award of ***Bachelor of Technology*** degree in ***Ceramic Engineering*** at National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance. Certified further, that to the best of my knowledge the work reported does not form part of any other thesis or dissertation on the basis of which a degree or award was conferred on an earlier occasion on this or any other candidate.

Date: 11 May, 2012

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ABSTRACT

Graphene, a monolayer form of carbon with two dimensional honeycomb lattices (sp^2 hybridization) has shown excellent mechanical, electrical, thermal and optical properties. It is a promising component for many applications in the fields of electronics, composites, sensors as well as energy storage and conversion devices. These engineering applications require availability of graphene on the mass scale and thus suitable processes are necessary for its production down to single sheet level. The present work focuses on the preparation of graphene on a bulk scale from graphite in an affordable and easy technique and the characterization of the graphene produced to test its quality. A report on the study of graphene by Raman spectroscopy, Scanning Electron Microscopy, Fourier Transform Infrared Spectroscopy and X-Ray Diffraction analysis is given here.

LIST OF FIGURES

LIST OF ABBREVIATIONS

Sl no	Abbreviation	Full Form
1	GIC	Graphite Intercalation Compounds
2	TEM	Transmission Electron Microscopy
3	GO	Graphite Oxide
4	SEM	Scanning Electron Microscopy
5	XRD	X-Ray Diffraction
6	FTIR	Fourier Transform InfraRed
7	AFM	Atomic Force Microscope
8	NMR	Nuclear Magnetic Resonance
9	XPS	X-ray Photoelectron Spectroscopy
10	FLG	Few Layer Graphene
11	SLG	Single Layer Graphene
12	HOPG	Highly Oriented Pyrolytic Graphite
13	CNT	Carbon Nano Tube
14	MWNT	Multi Wall carbon Nano Tube
15	DI	De-Ionized

CONTENTS

1. INTRODUCTION

[1.1] GRAPHENE

Graphene is a single layer of carbon atoms (cf. Fig.1) which is bonded together in a hexagonal lattice. The honeycomb lattice consists of two equivalent sub-lattices of carbon atoms bonded together with σ bonds. This material has led to a vast amount of research in recent years [1].

The term “graphene” first appeared in 1987 to describe single sheets of graphite as one of the constituents of graphite intercalation compounds (GICs); conceptually a GIC is a crystalline salt of intercalate and graphene [2]. The term was also used in early descriptions of carbon nanotubes, as well as for epitaxial graphene, and polycyclic aromatic hydrocarbons.

Single layers of graphite were grown epitaxially on top of other materials. This "epitaxial graphene" consists of a single-atom-thick hexagonal lattice of sp^2 bonded carbon atoms, as in free-standing graphene [3]. However, there is significant charge transfer from the substrate to the epitaxial graphene, and, in some cases, hybridization between the d orbital of the substrate atoms and π orbital of graphene, which significantly alters the electronic structure of the epitaxial graphene. Single layers of graphite, observed by TEM within bulk materials, in particular inside soot was obtained by chemical exfoliation.

A key advance in the science of graphene came when Andre Geim and Konstantine Novoselov at Manchester University managed to extract single-atom-thick crystallites (graphene) from bulk graphite in 2004. The Manchester researchers pulled out graphene layers from graphite and transferred them onto thin SiO_2 on a silicon wafer in a process sometimes called **micromechanical cleavage** or, simply, the **scotch tape technique** [4]. On October 5, 2010, the

Nobel Prize in Physics for the year was awarded to Andre Geim and Konstantin Novoselov from the University of Manchester for their work on graphene [5].

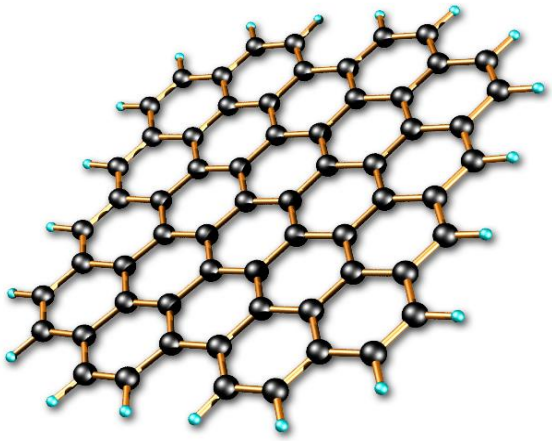

Fig.1. Monolayer graphene[#]

A few properties of graphene which makes it special are [6, 7-9]:

- It is the purest form of carbon
- Large theoretical specific area (2360 m^2/g)
- High intrinsic mobility (200,000 $cm^2v^{-1}s^{-1}$)
- Extremely high Young's modulus (~1.0 TPa)
- Thermal conductivity (~5000$Wm^{-1}K^{-1}$)
- Optical transmittance (~97.7%)

Graphene has a robust and flexible membrane, which provides essentially infinite possibilities for the modification or functionalization of its carbon backbone.

[1.2] Methods of graphene synthesis

Various processes available to prepare graphene are:

- Drawing method
- Epitaxial growth on silicon carbide

http://www.zeitnews.org/chemistry-physics-and-material-sciences-research/new-10-year-billion-euro-plan-for-graphene.html

- Epitaxial growth on metal substrates
- Growth from metal-carbon melts
- Pyrolysis of sodium ethoxide
- Unzipping of carbon nanotubes
- From graphite by sonication (Delamination process)
- Dry ice method
- Micromechanical cleavage
- Heat treatment of silicon wafers.
- CVD in controlled conditions with temperature gradient, etc.
- **From Graphite**

[1.3] Few areas of application of graphene

Graphene has excellent thermal, electrical, mechanical and electronic properties.

The areas of importance are

- Single molecule gas detection
- Graphene transistors
- Transparent Conducting Electrodes
- Heat dissipation systems
- T-ray scanners
- Ultra capacitors
- Flexible solar cells
- Integrated circuits
- Creation of multi wall nanotubes and many other allotropes of carbon
- Anti microbial for purification of water in drinking

- Bio-devices
- Electro chromic devices
- Polymers and composites

[1.4] GRAPHITE OXIDE (GO)

Graphite oxide (GO) is a hydrophilic derivative of graphene. Surface bound oxidized functional groups contribute to the hydrophilic properties of graphene [10]. GO is produced by the oxidative treatment of graphite by one of the principal methods developed by Brodie, Hummers or Staudenmeir [11-14]. GO consists of graphene sheets decorated mostly with epoxide and hydroxyl groups [15]. Rapid heating of GO results in expansion and delamination, due to evaporation of the intercalated water and evolution of gases from pyrolysis of the oxygen containing functional groups. By nature, GO is electrically insulating and cannot be used, without further processing, as a conductive material. In addition, the presence of the oxygen functional groups makes GO thermally unstable. Notably, it has been demonstrated that the electrical conductivity of GO (and presumably its thermal stability as well) can be restored close to the level of graphite by chemical reduction using hydrazine hydrate. The structure and properties of graphite oxide depend on the synthesis method and the degree of oxidation. However detailed structure is still not understood due to strong disorder and irregular packing of layers. The synthesis of graphene from graphite flakes is represented in figure 2.

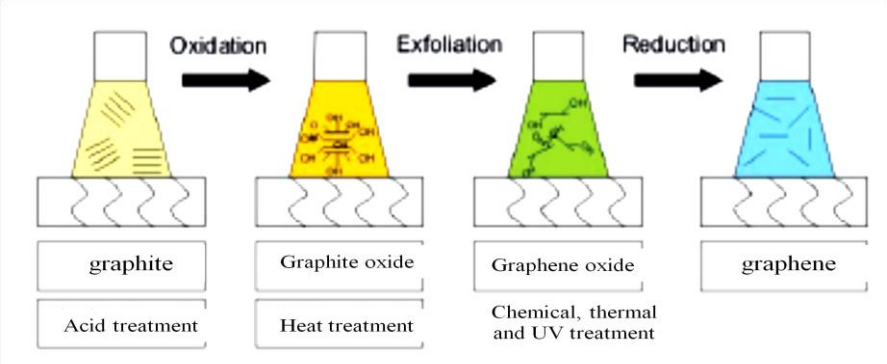

Fig.2. Process flow from graphite to graphene[#]

UrszulaKosidlo et al : Production methods of graphene and resulting material properties

2. LITERATURE REVIEW

Stankovich et al (2007) [15] synthesized graphene-based nano sheets via chemical reduction of exfoliated graphite oxide, performed through colloidal suspension route. GO was prepared from natural graphite by the Hummers method. GO (100 mg) was loaded in a 250 ml round bottom flask and water (100 ml) was then added, which yielded an inhomogeneous yellow-brown dispersion. This dispersion was sonicated using a Fisher Scientific FS60 ultrasonic bath cleaner (150 W) until it became clear with no visible particulate matter. Hydrazine hydrate (1.00 ml) was then added and the solution was heated in an oil bath at 100 $^{\circ}$C under a water-cooled condenser for 24 h over which the reduced GO gradually precipitated out as a black solid. This product was isolated by filtration over a medium fritted glass funnel, washed copiously with water (500 ml) and methanol (500 ml), and dried on the funnel under a continuous air flow through the solid product cake. The product was characterized with SEM, Raman, XPS, NMR spectroscopy, TGA, elemental analysis and electrical conductivity. Reduced GO characterizations indicated the formation of unsaturated and conjugated carbon atoms, which imparted electrical properties of graphene sheets. Thus, reduced graphene oxide sheets may be used in hydrogen storage devices and as an electrically conductive filler material in composites.

Kaniyoor et al (2010) [16] synthesized graphene at low temperature by exfoliating graphitic oxide in hydrogen atmosphere. They followed Modified Hummers method for the production of GO using SP-1 graphite. Graphite was mixed properly with NaCl and then washed with DI water. Filtration was performed and then the filtrate was stirred with conc. H_2SO_4 for 8 h. 6g of $KMnO_4$ was added gradually, keeping the temperature less than 20 °C. The mixture was stirred

at 35 °C for 30 minutes and at 65 °C for 40 minutes. 92 ml water was added to the solution prepared and it was heated to ~100 °C. The concentration of the solution was reduced by addition of 280 ml of water and then 30% H_2O_2. The mixture was centrifuged and filtered which constituted the washing process (5% HCl initially and then water). It was followed by washing, and then vacuum drying. The powder, thus produced, was characterized with XRD, FTIR, FESEM, Raman spectroscopy, BET, DSC, and XPS.

Marcano et al (2010) [17] synthesized graphene oxide through an improved route**.** Graphite flakes was oxidized using $KMnO_4$ and 9:1 mixture of conc. H_2SO_4 and conc. H_3PO_4. The product was characterized by Raman, FTIR, AFM, TEM, XPS, NMR etc. and properties were compared between products synthesized from Hummers and Modified Hummers method. The "improved graphene oxide" showed regularity in its structure with a greater amount of retention of basal plane framework and less defects in the framework. Other advantages of this method are high productivity, no toxic gas evolution during preparation, and equivalent conductivity upon reduction, making it a good and reproducible approach for preparing material on a bulk amount.

Hernandez et al (2009) [18] synthesized graphene in liquid phase by exfoliation of graphite in surfactant/water solution. The fine graphite powder was separated from large particles using a mesh. A different concentration of sodium dodecylbenzene sulphonate (SDBS) solutions was prepared in millipore water, stirring it overnight. Graphite powder was dispersed in the SDBS solutions (in 25 ml cylindrical vials), followed by sonication for half an hour. The dispersed solution was left undisturbed for 24 h for the formation of unstable aggregates. After that, it was centrifuged for 90 minutes at 500 rpm. 15 ml, from the top of the vial was used after decantation. Various characterizations were performed which showed the formation of few layer grapheme (FLG).

Chen et al (2009) [19] synthesized graphene oxide from expanded graphite in presence of a strong oxidant. The expanded graphite powder (20 g) was put into concentrated H_2SO_4 (460 ml) followed by gradual addition of $KMnO_4$ (60 g) with simultaneous stirring and cooling, keeping the temperature of the mixture below 20 °C. The mixture was then stirred at 35 °C for two hours with the addition of deionized water (920 ml). The reaction was terminated within one hour by the addition of DI water (2.8 l). 30% H_2O_2 solution (50 ml) was addition caused violent effervescence with an increase in temperature to 100 °C and the color of the suspension changed to bright yellow. The suspension was washed with 1:10 HCl solution (5 l) to remove metal ions by filter paper and funnel. The paste collected from the filter paper was dried at 60 °C, until it again formed agglomerates. The agglomeration was dispersed in deionized water in static state for 2 to 3 h and slightly stirred by glass bar. The suspension was then washed with deionized water for 5 to 7 times for two days by filter paper and funnel, until the pH became 7. The paste, collected on the filter paper was dispersed into water by ultrasonication. The obtained brown dispersion was then subjected to 30 minutes of centrifugation at 4000 rpm. The GO platelets were obtained by dehydration at 60 °C in air. Various characterizations were then performed to test the quality of graphene oxide produced. This study highlights the approach to large quantity preparation of graphene oxide in industry via an inexpensive and simple method.

Chung (1987) [20] threw some light on the process of exfoliation of graphite and exfoliated graphite material. The exfoliation of graphite is a process in which graphite expands up to hundred times along the c-axis, resulting in the formation of a puffed up material with low density and high temperature resistance. Exfoliated graphite serves the purpose of lubrication when it is compressed and also develops flexibility. Exfoliation of graphite can be done by

internal heating, external heating or by excessive electrolytic intercalation. This study enlightens us about the various applications of exfoliated graphite.

Synthesis techniques of Graphene:

The drawing method uses a piece of cellophane tape to draw a thin film from highly oriented pyrolytic graphite. After repeated peeling from the thin film, it is ultimately stamped onto a substrate and the tape is carefully amputated. The deposition formed is a dense network of single and multi-layered graphene, which is cleansed before using for specific purpose [21].

Epitaxial graphene on silicon carbide process is used to prepare graphene by heating followed by cooling down a SiC crystal. We can produce epitaxial graphene with dimensions depending upon the size of the SiC substrate (wafer). Similar to this, is another popular techniques of graphene growth known as ***thermal decomposition of Si*** on the (001) surface plane of single crystal of 6H-SiC. 1 to 3 graphene layers are formed due to epitaxial growth, the number of layers being dependent on the decomposition temperature [22].

Epitaxial growth on metal substrates is another commonly used method for the synthesis of graphene. The metals used as substrates show catalytic activity for hydrocarbon decomposition at high temperature. The growth mechanism follows the principle of segregation of carbon, carbon vapor deposition or by chemical vapor deposition which involves hydrocarbon decomposition on the catalytic metal surface. Graphene is also grown by exposing a Ni film to a gas mixture of H_2, CH_4 and Argon at 1000 °C. The average number of layers depends on the Ni thickness. Moreover, the shape of the graphene depends on patterning of the Ni layer.

The prime mechanism ***in growth from metal-carbon melts process*** is to dissolve carbon atoms inside a transition metal melt at a certain temperature, and then allowing the dissolved carbon to precipitate out at lower temperatures as single layer graphene (SLG). Another process is the

reduction of ethanol by sodium metal, followed by pyrolysis of the ethoxide product and washing with water to remove sodium salts.

Another new method of graphene synthesis has utilized multiwall carbon nanotubes (MWNT) as the starting material. The process is popularly known as **'*un-zipping* of CNTs'**. Nanotube and polymer are spun to create a film in which the polymer covers up part of the tubes [22]. The film is then peeled away and exposed to argon plasma in a reaction chamber, which etches away the exposed carbon atoms. The size of the resulting graphene nanoribbons can be controlled by varying the thickness of the polymer film and the reaction times involved.

In micromechanical exfoliation method, graphene is disaffiliated from a graphite crystal using adhesive tape. After peeling it off the graphite, multiple-layer graphene remains on the tape. By this process of peeling the multiple-layer, graphene is cleaved into various flakes of few layers graphene. The tape is then attached to the substrate and the glue solved, e.g. by acetone, in order to detach the tape. Finally one last peeling with an unused tape is performed.

3. EXPERIMENTAL PROCEDURE

[3.1] Purification of graphite flakes:

Chemical purification is designed to remove impurities that exist or are "intercalated" between the various graphite planes. This type of treatment is critical for improving and ensuring the maximum electrical conductivity in applications such as battery grade materials. As the name implies flake graphite has a distinctly flaky or platy morphology. All graphite has a unique flaky morphology on some level, but in most instances flake-graphite has the same structure irrespective of particle size.

For purification of the graphite flakes, first of all, 30 ml of hydrofluoric acid was carried off in a plastic beaker. Then to it 2 g of graphite flakes were added. Then mechanical stirring was done to accomplish a homogeneous mixture. After that sufficient time was provided to settle the graphite flakes in the mixture. When settling of graphite flakes was complete, pH of the same was measured. Because of the acid treatment pH was found in the acidic zone. So to it distilled water was added continuously, so that the acid content could be washed off. The same procedure was continued for several times until the pH became neutral. Then ultrasonication was done to achieve proper mixing of the graphite flakes with liquid and after that it was again allowed to settle down and decant the liquid. Later acetone was added to the dispersion and it was dried in an oven at around 100 °C. Pure graphite flakes were obtained by this process.

The layout of purification of graphite flakes has been presented and explained in figure 3.

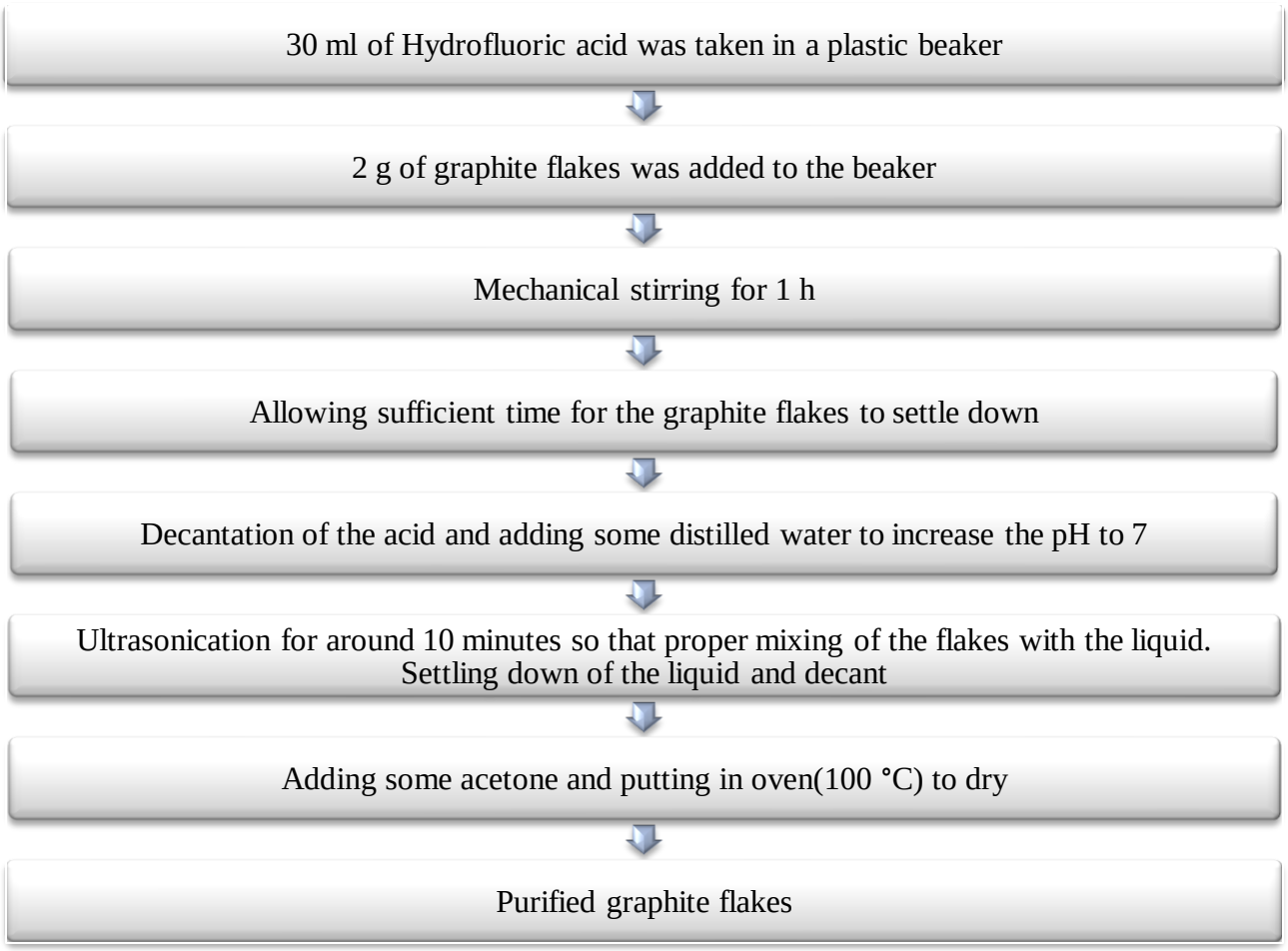

Fig.3. Flowchart showing purification of graphite flakes

[3.2] Synthesis of Graphite oxide using modified Hummer's Method:

Graphite oxide was prepared from natural graphite by a modified Hummers method. Briefly, graphite powder (2 g; 97% purity; IFGL Refractories) and $NaNO_3$ (1 g; Aldrich, >99%) were mixed, then it was put into concentrated H_2SO_4 (96 ml; Fisher Scientific, 98%) with an ice bath. Under vigorous stirring, $KMnO_4$ (6 g; Fisher Scientific, 99.6%) was added gradually. The temperature of the mixture was maintained below 20 °C. After removing the ice bath, the mixture was stirred at 35 °C in a water bath for 18 h. As the reaction extended, the mixture turned out to be pasty with a brownish color. Successively, 150 ml of H_2O was moderately added

to the paste. Addition of water into the concentrated H_2SO_4 medium releases a large amount of heat; therefore, to maintain the temperature below 50 °C, water was added continuously. After dilution with 240 ml of H_2O, 5 ml of 30% H_2O_2 (Fisher Scientific) was added to the mixture, and the diluted solution color transformed to brilliant yellow along with bubbling. After continuous stirring for 2 h, the mixture was filtered and washed with 10% HCl aqueous solution (250 ml; Fisher Scientific), de-ionized water, and ethanol (Fisher Scientific; anhydrous) to remove other ions. The resulting solid was dried by vacuum.

The whole procedure for synthesis of graphite oxide has been cited in the figure 4.

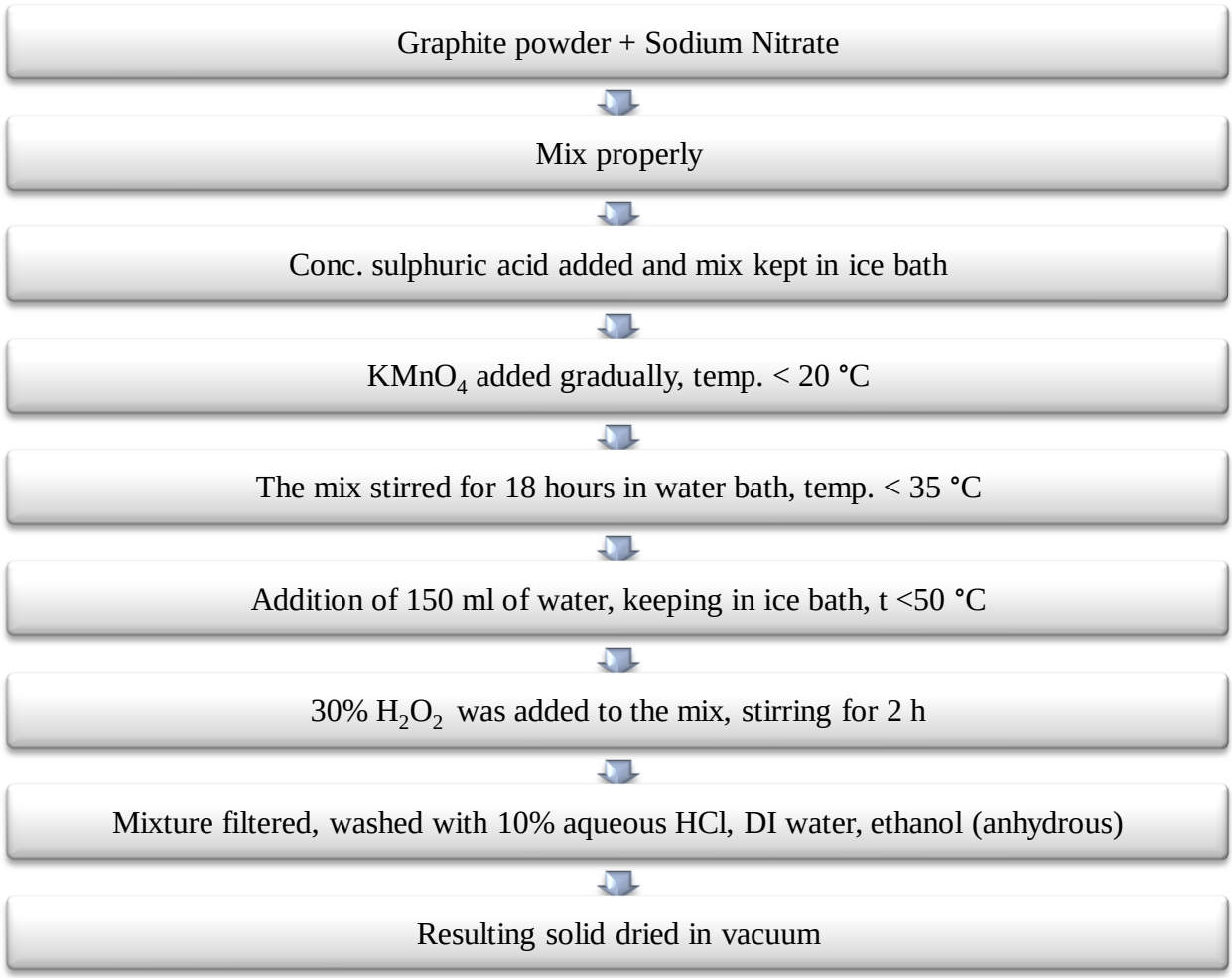

Fig.4. Flow chart showing the synthesis of graphite oxide

[3.3] Stabilization of GO suspension:

The most important factor of GO preparation in Hummer's method is the slow filtering rate. The residual acids are hardly removed. So the suspension was washed with 1:10 HCl solution to remove the suspended metal ions. However during the drying stage, complete removal in the acids from the GO is not possible. Thus an attempt was made to increase the pH of the solution by washing with de-ionized water which resulted in the formation of colloid. The reason to increase the pH of the suspension is to stabilize GO. Higher will be the pH, larger will be the zeta potential of GO as a function and the electrostatic force among GO sheets. If the electrostatic force among GO sheets exceeds or becomes equal to the total of gravity and van der Waals force, the GO hydrosol attains stability. The stabilization process of GO sheets is represented in figure 5.

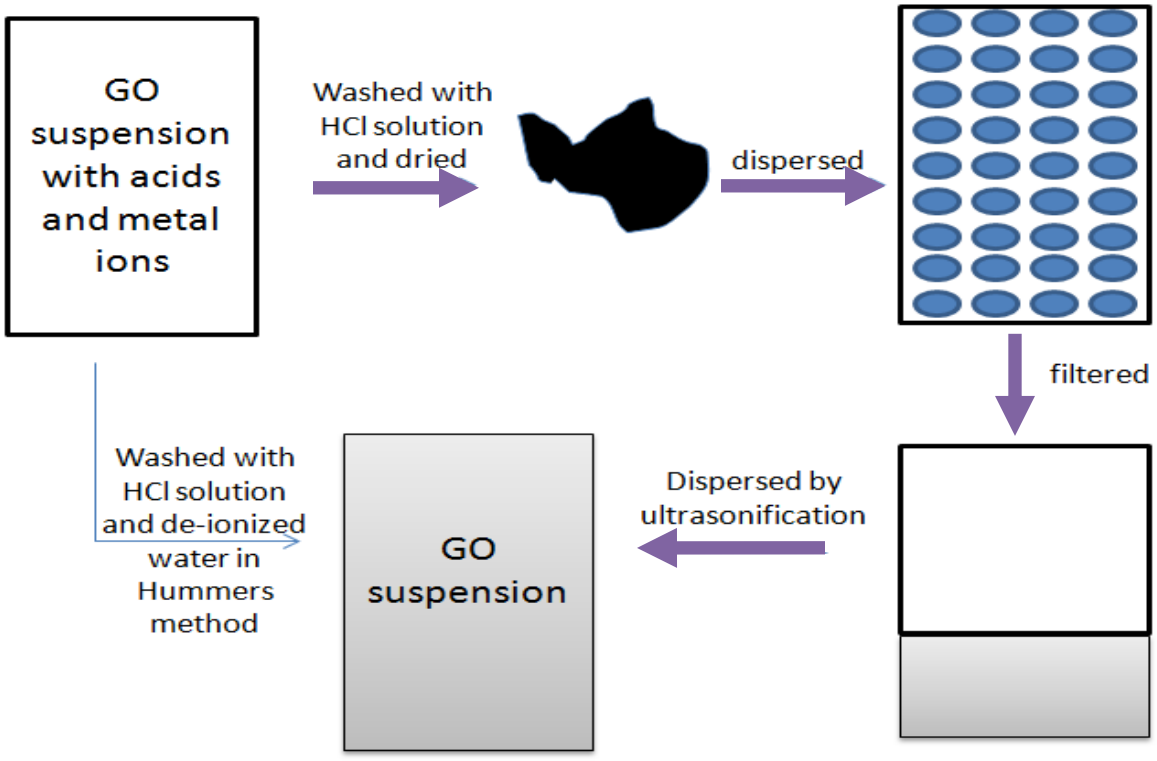

Fig.5. different process between modified Hummers method and Hummers method

[3.4] Reduction of graphite oxide to graphene:

Pre-calculated amount of graphite oxide (2 g) powder was mixed with 500 ml of water in a beaker and sonicated vigorously for 30 minutes to prepare the colloidal solution. 10 ml of hydrazine hydrate was added to the solution and poured into the soxhlet apparatus mantle. The round bottom flask was set to heating at 100°C, set in a water cooled condenser for 24 hours, over which the reduced graphite oxide gradually precipitated out as a black solid. The product was isolated by filtration and washed copiously with water and dried. The reduction process is represented in figure 6.

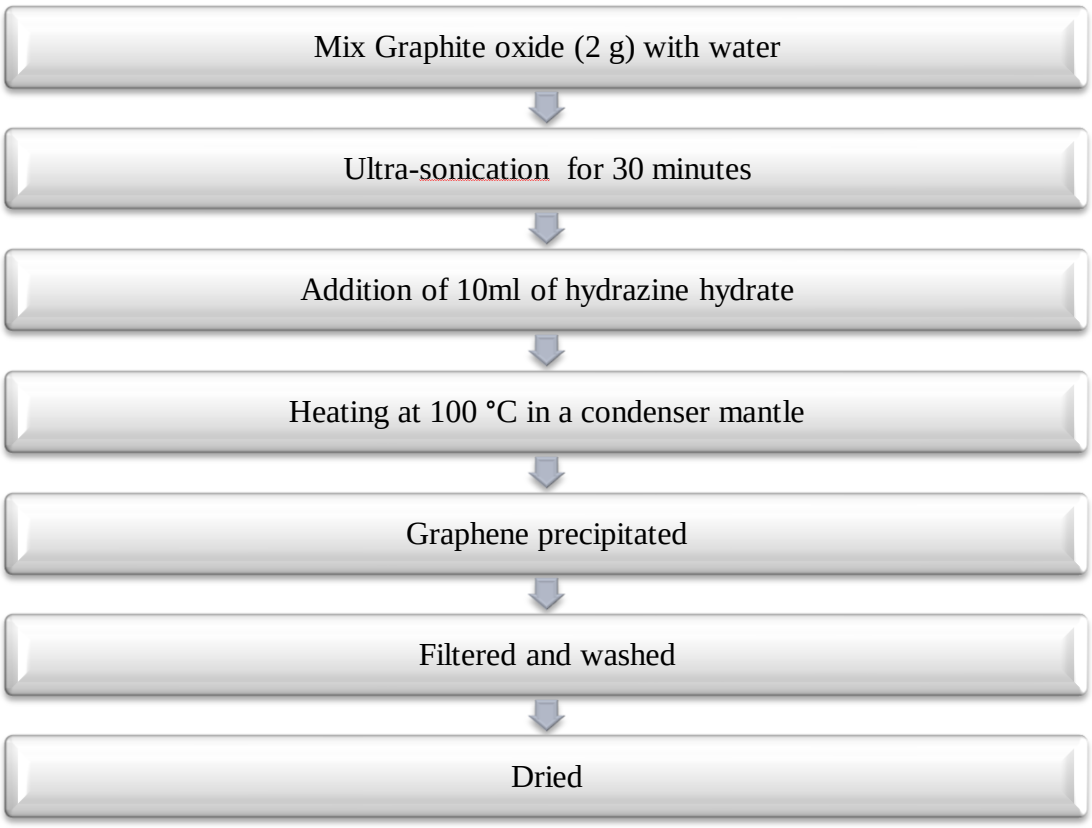

Fig.6. Flow chart showing the reduction of graphite oxide to graphene

4. CHARACTERIZATION

[4.1] X-Ray Diffraction:

X-ray diffraction is a technique whichcontributes detailed information about the chemical composition and crystallographic structure of the materials. It is also a unique tool used to detect the presence of phases in the material.

The main principle behind XRD is Bragg's law which states

$$n\lambda = 2d\sin\theta$$

Where, d = spacing between diffracting planes,

θ = incident angle,

n = any integer

λ = wavelength of the beam

In this work, XRD analysis was performed by Philips' X-ray diffractometer with Nickel filtered Cu Kα radiation (1.5406Aº). The diffraction was done at angle 10°-60º with scanning speed 2º per min.

[4.2] Raman spectroscopy:

Raman spectroscopy is employed to study various samples. It is a technique based on inelastic scattering of monochromatic light, where the sample absorbs photons of the laser lightand then re-emits causing a transition. Frequency of the reemitted photons shifts up or down with respect to original monochromatic frequency. This is called the Raman Effect. This shift or the transition provides sufficient information about rotational, vibrational and other transitions occurring in molecules.

A sample is normally irradiated with a laser beam in the ultraviolet, visible or near infrared range. Scattered light is collected with a lens and is sent through interference filter or spectrophotometer to obtain Raman spectrum of a sample. A simple set up and raman states is represented in figure 7 and 8

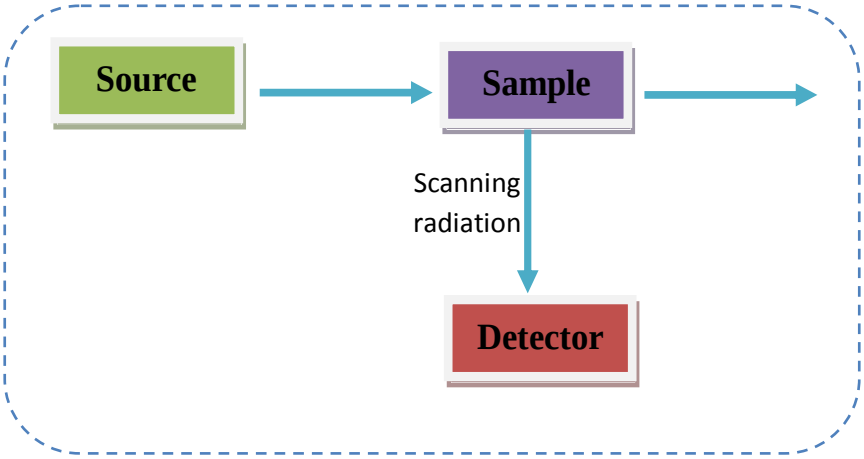

Fig.7. Basic set up of a Raman spectroscopy

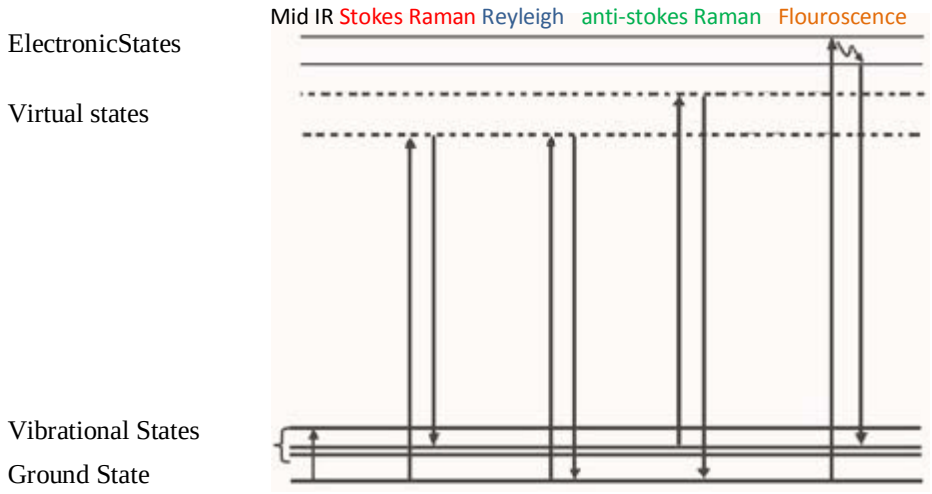

Fig.8. Raman transitional states[#1]

[4.3] Fourier Transform InfraRed Spectroscopy:

Fourier transform infrared spectroscopy (FTIR) is a technique adopted to obtain an infrared spectrum of absorption, emission, and photoconductivity of a solid, liquid or gas. Also it can be utilized to quantitative analysis of an unknown mixture. By adapting the infrared

#1 Raman Spectroscopy/ en.wikipedia.com

absorption spectrum, the chemical bonds in a molecule can be identified. FTIR spectrumis a kind of molecular "fingerprint" because of its uniqueness. It works upon the basic principle of Beer-Lambert or Beer's Law. It's a straightforward study of the absorption process of photons which passes through an absorbing medium. It assumes the intensities of the peaks are directly related to the amount of sample present.

$$A = \log_{10}[I_0/I] = \text{Ɛ}bC$$

Where, I = intensity

Ɛ= molar absorptivity (molar extinction co-efficient)

B= path length

C= concentration

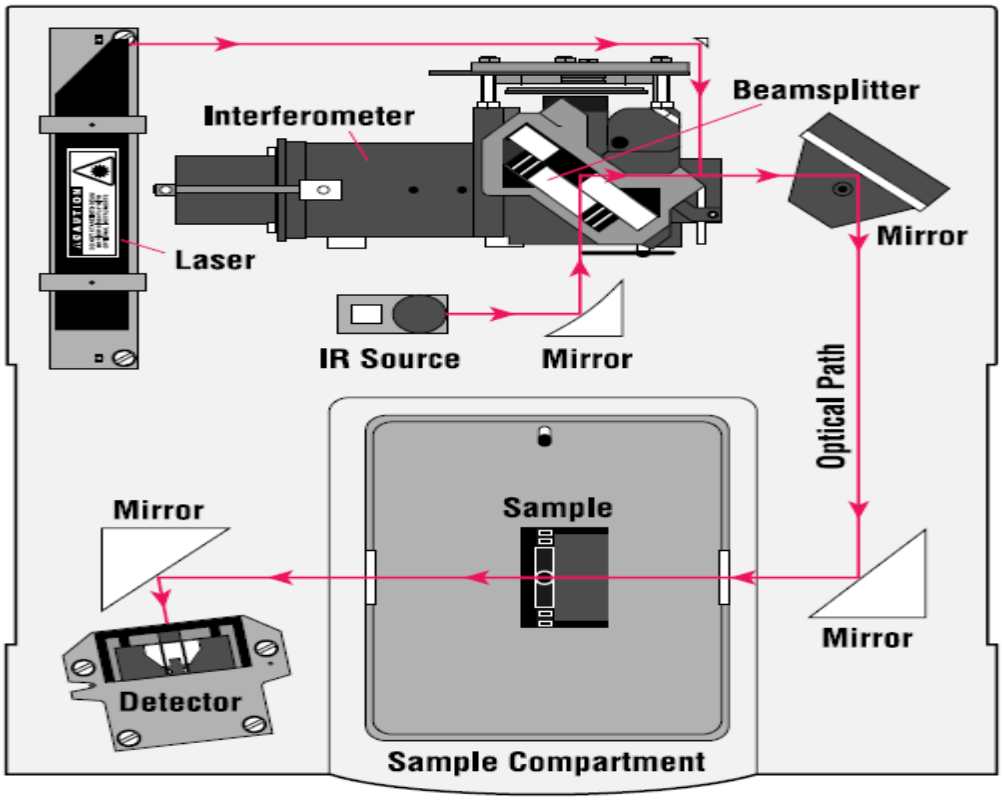

Fig.9. set up of FTIR Spectroscopy[#2]

2 Princeton Instruments, Raman Spectroscopy basics/ www.pricetoninstruments.com

Various sampling techniques for FTIR are transmission, attenuated total reflection, specular reflectance, diffuse reflectance, diffuse reflectance Fourier transform spectroscopy, polarization modulation infrared reflection adsorption spectroscopy, portable FTIR etc.

For FTIR spectroscopy test powder sample was mixed with KBr to form pellets. As KBr crystal has no visible peaks in the infrared spectrum, so that the peaks corresponding to the powder sample can be analyzed. Then obtained peaks were matched with the corresponding bonds, bending vibrations etc. and sample was analyzed.

[4.4] Scanning Electron Microscopy:

The scanning electron microscope (SEM) is operated to extract information about external morphology (texture), chemical composition, and crystalline structure and orientation of materials.The SEM assays specific locations on the sample, and helps in qualitatively or semi-quantitatively determining chemical compositions (using EDS), crystalline structure, and crystal orientations (using EBSD).

Using conventional SEM techniques approximately 1 cm to 5 microns in width (of area) can be imaged in a scanning mode. Upon increasing the magnification clear image of the sample is obtained.

5. RESULTS AND DISCUSSIONS

[5.1] X-Ray Diffraction analysis:

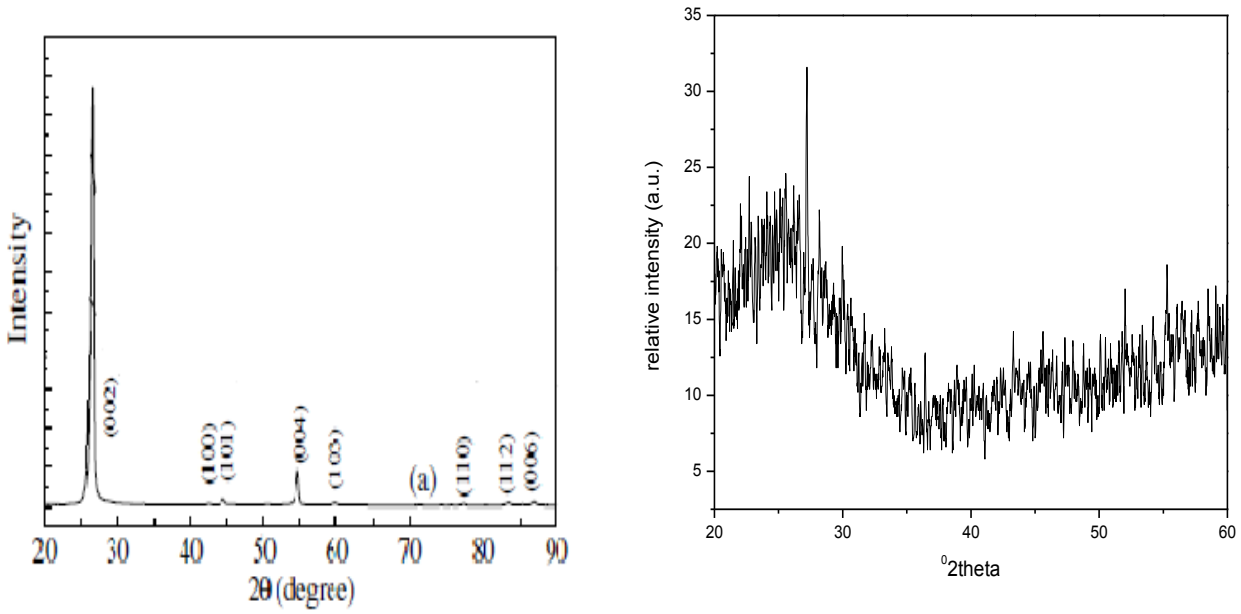

Fig.10. XRD pattern of graphite* (left) and graphite oxide (right, current study)

Figure 10 represents X-Ray diffraction patterns of graphite oxide and graphene respectively. Graphite contains a very distinct and sharp (002) peak which transforms into a broad shaped peak in graphite oxide due to addition of the functional groups. Graphite loses its crystallinity and converts into a semi-crystalline and hydrophilic structure on oxidation.

*Z.Q. Li, C.J. Lu, Z.P. Xia, Y. Zhou, Z. Luo, "X-ray diffraction patterns of graphite and turbostratic carbon" Carbon 45 (2007) 1686- 1695

[5.2] Raman Spectroscopy analysis:

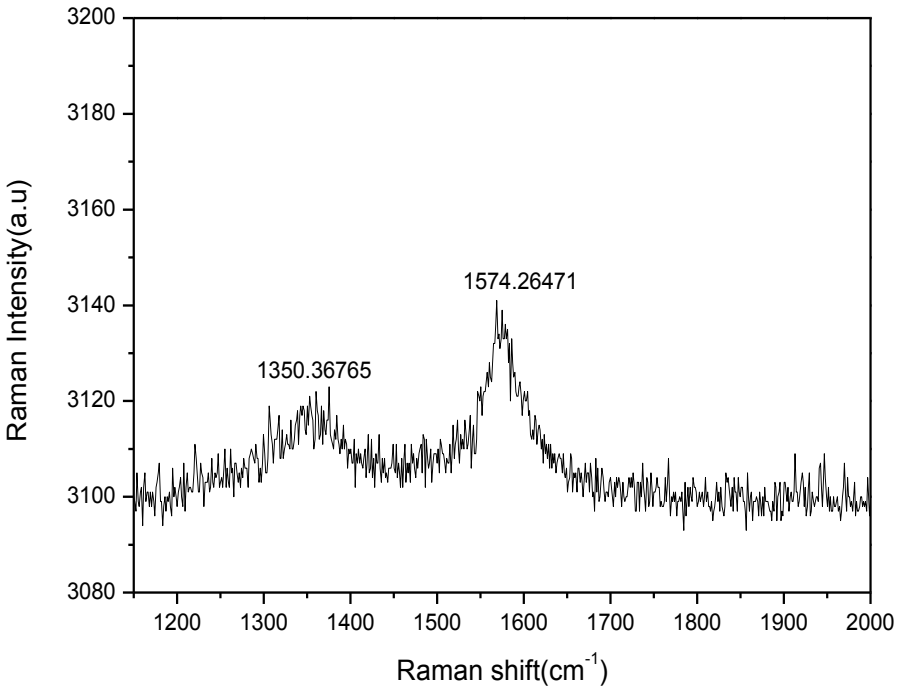

Fig.11. Raman spectrum of graphene

Figure 11 shows Raman spectrum of graphene. In Raman spectroscopy, D band indicates the extent of defects whereas G band indicates the graphitic nature respectively. If the D peak intensity is higher, the sample is having more defects in graphite network. These defects will be imparted to the edges of the sheets. If the G band is prominent, sample is said to be crystalline. D band occurs at 1350 cm^{-1} and G band occurs at 1574 cm^{-1}. Presence of D band and G band confirms the formation of graphene with lesser defects and crystalline structure.

[4.3] Scanning Electron Microscopy analysis:

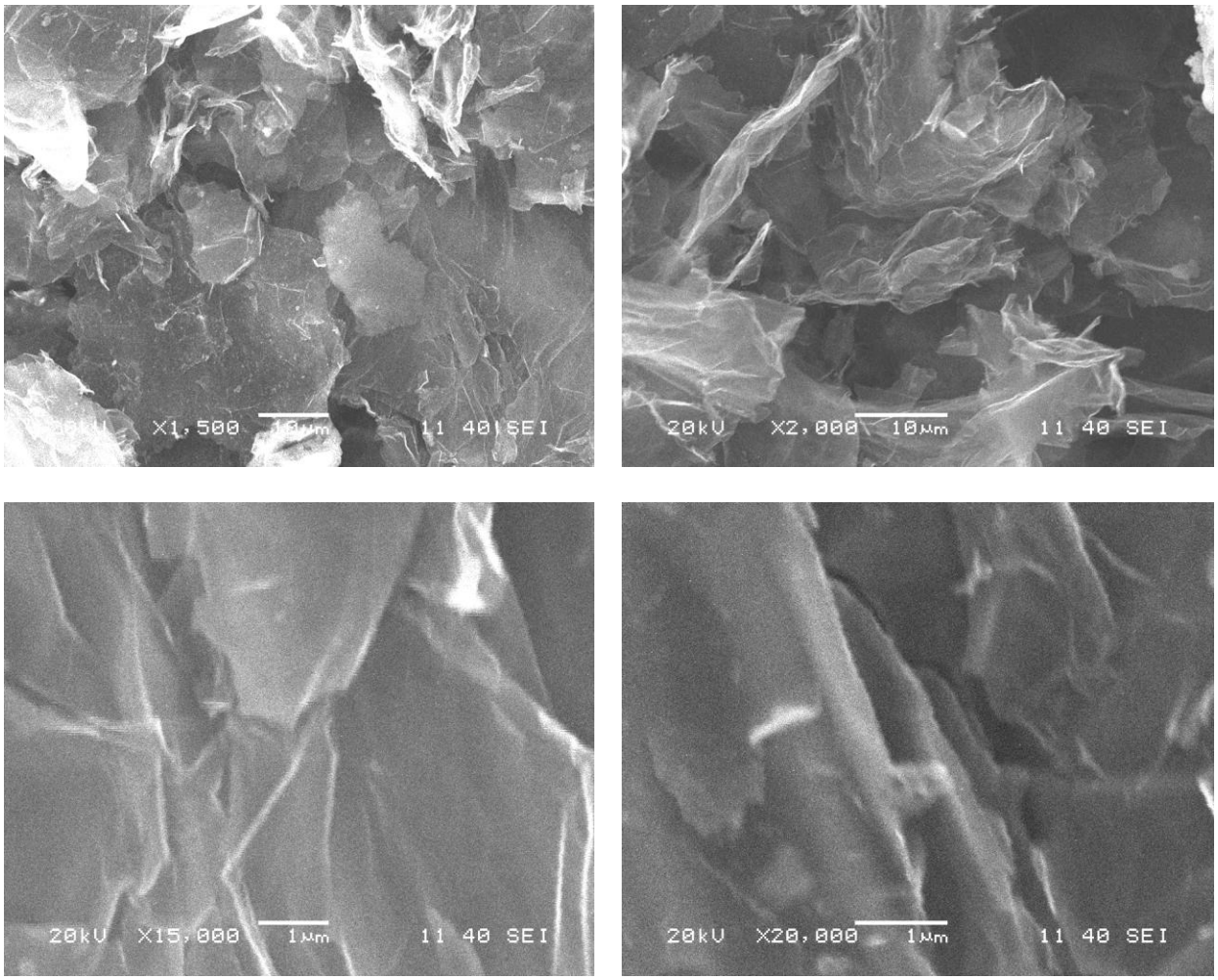

Fig.12. SEM micrographs of graphite oxide platelets

Figure 12 shows images of graphite oxide, which was prepared directly from its graphite platelets and without exfoliating small clusters of graphite oxide sheets. Graphite oxide consists of layered structure of graphene oxide sheets that are strongly hydrophilic and intercalation of different functional groups between the layers occurs readily. The random orientation and wavy appearance of exfoliated graphite oxide is seen from the SEM images.

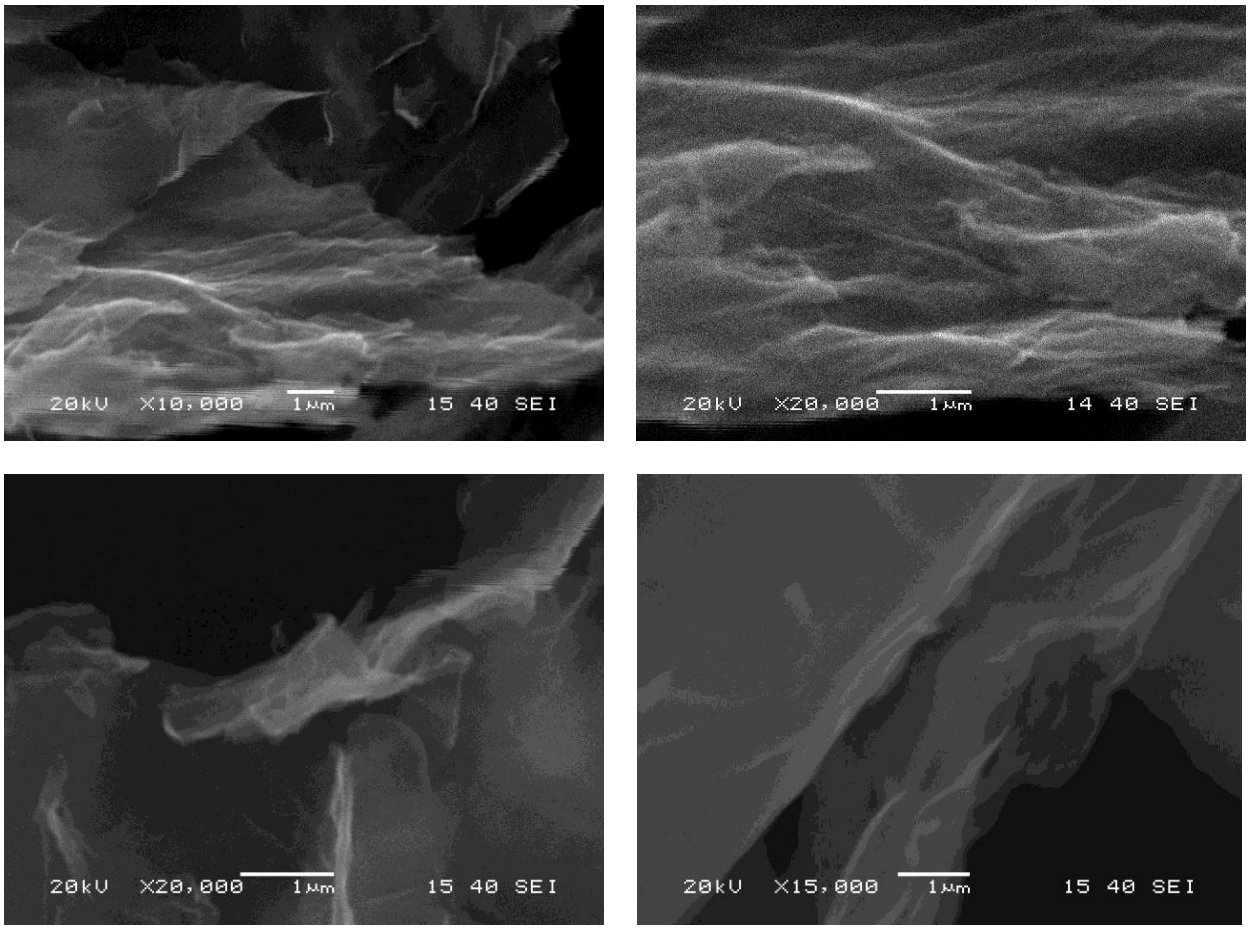

Fig.13. SEM micrographs of reduced graphite oxide sheets

Figure 13 show reduced graphite oxide having isolated layers, which are nothing but graphene. The graphene sheets formed showed wrinkled appearance and appear to be very thin. It can be explained that during reduction, hydrazine has readily reacted with the epoxide functional groups to form hydrazine alcohols, which are mainly responsible for the incorporation of nitrogen. The graphene layers are more or less transparent as observed from these images.

[4.4] Fourier Transform Infrared spectroscopy:

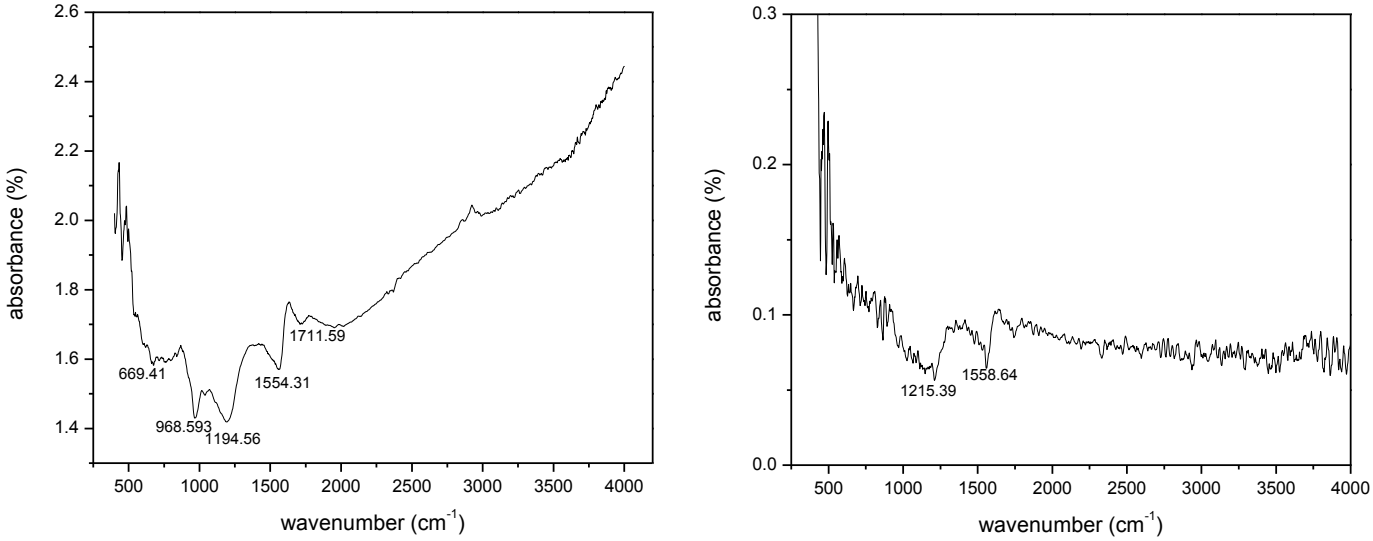

Fig.14. FTIR spectra of graphite oxide (left) and graphene (right)

Figure 14 represents FTIR spectra of graphite oxide and graphene oxide respectively. For graphite oxide, aromatic content appears at 669.40 cm^{-1}, epoxy groups at 968.59 cm^{-1} and 1194.56 cm^{-1}, C=C stretching at 1554.31 cm^{-1}, C=O and C-O stretching at 1711.59 cm^{-1}. For graphene, epoxy content at 1215 cm^{-1}, C=C stretching at 1558.63 cm^{-1}. On reduction, the functional groups disappear as evident from the above plot.

6. SUMMARY

1. Bulk synthesis of graphene nanoplatelets could be carried out using refractory grade graphite as the starting material.
2. The method followed was easy, environment friendly, and cost effective.
3. Scanning Electron Microscopy showed fluffy morphology and few layer of graphene sheets.
4. Raman spectroscopy showed the evidence of the formation of graphene with the appearance of D and G peaks.
5. It is expected that relatively purer form of graphite (higher refractory grade) precursor will enhance the quality of graphene nanosheets.

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